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SOLID STATE COEXTRUSION OF HIGH DENSITY POLYETHYLENE. II. EFFEC--ETC(U)
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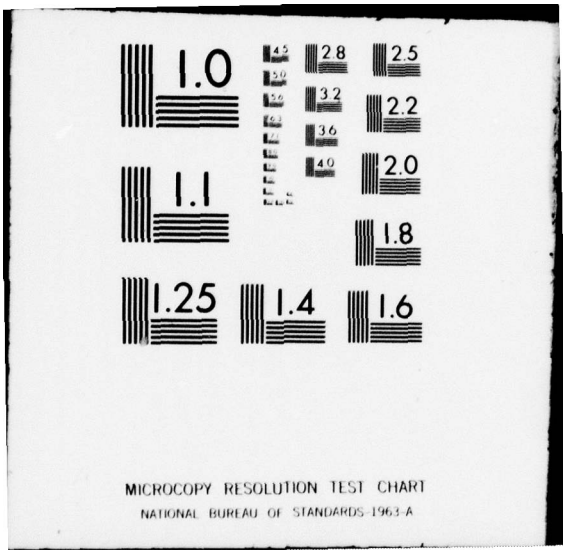
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NATIONAL BUREAU OF STANDARDS-1963-A

(12)

OFFICE OF NAVAL RESEARCH

(15) Contract No. N00014-75-C-0686

Project No. NR 356-584

(14) TR-11

(9) TECHNICAL REPORT, NO. 11

(6) SOLID STATE COEXTRUSION OF HIGH DENSITY POLYETHYLENE. II. EFFECT OF MOLECULAR WEIGHT AND MOLECULAR WEIGHT DISTRIBUTION

by

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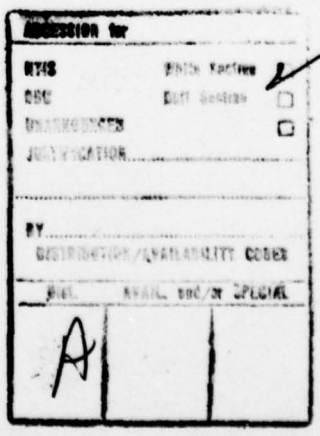
(12) 34 p.

(11) 16 October 16, 1978

ABSTRACT

The recently developed technique of solid state coextrusion for ultradrawing semicrystalline thermoplastics has been applied in the preparation of self-reinforced high density polyethylene extrudates. The extrudates consist of definite core and sheath phases composed of different molecular weights (M_w) in the range of 60,000 - 250,000 and different molecular weight distributions (M_w/M_n = 0.3 - 20). Cocylindrical billets of two different phases were prepared for extrusion by inserting a polyethylene rod within a tubular billet of a different high density polyethylene followed by melting the two phases to obtain bonding between them. The billet was then split longitudinally to increase extrusion speed followed by extrusion at 120°C., 0.23 GPa and extrusion draw ratio 25.

Thus it was possible to produce extrudates of high tensile modulus (45 GPa) and strength (0.55 GPa) at a rate near 0.7 cm./min. In general, the tensile properties of the extrudates increased with average molecular weight and were insensitive to the molecular weight distribution of the two phases.



INTRODUCTION

The development of high strength forms of semicrystalline thermoplastics has been pursued recently with considerable interest by employing essentially three different techniques: crystalline state extrusion, drawing and drawing from solution[1-8].

Crystalline state extrusion and drawing deal with the solid state deformation of thermoplastic polymers (polyethylene being the primary candidate). There are several parameters which may influence the extrusion or drawing behavior. Two such parameters are the molecular weight (M_w) and molecular weight distribution (M_w/M_n). Ward and his co-workers[3-5] have reported on the effects of these parameters on drawing. They report that molecular weight and distribution affect the draw rate and impose a limit to the maximum obtainable draw ratio for specific drawing conditions (temperature and strain rate). Porter et al.[9] in their studies of the effect of molecular weight on the mechanical properties of ultradrawn high density polyethylene (HDPE) report that the higher molecular weight polyethylenes give higher strength fibers yet with no apparent effect on the Young's modulus which may have been concealed by annealing effects. In addition, solid state extrusion of high molecular weight polyethylenes at lower temperatures, <120°C., where annealing does not occur proceeds slowly for extrusion draw ratios >15 and hinders the systematic study of the effect of molecular weight. This limitation has been partially alleviated by the recently developed technique of solid state coextrusion[10,11].

In previous publications[12,13] we reported the results of the extrusion of self reinforced thermoplastic composites and emphasized the feasibility of crystalline coextrusion of polyethylenes of different molecular characteristics. We also reported that the extrusion rate was faster when the high molecular weight HDPE component was used as sheath rather than as core component in the initial preformed billet.

In this report we discuss the effects of the molecular parameters on the tensile properties and extrusion rate of solid state coextruded high density polyethylenes.

EXPERIMENTAL

(a) Preparation of Composite Billets

Composite billets of different high density polyethylenes were prepared in a specially designed apparatus[11,13]. Briefly, sheath components were prepared by drilling out preformed rods into tubular billets and core components were produced by turning down on a lathe a billet of the appropriate polyethylene to a diameter equal to the inner diameter of the drilled out billet. Subsequently, the sheath and core sections were melted together into one billet consisting of two different components. The volume fraction of the core component was equal to 25%. The composite billets were then split longitudinally into two semiperipheral segments which assembled side by side were then press-fitted into the reservoir of an Instron Capillary Rheometer and coextruded.

In these studies the composite billets were extruded at 120°C. and 0.23 GPa through a brass conical die of length 2.54 cm. and nominal extrusion draw ratio 25 defined as the ratio of die entrance to exit cross sectional areas.

Initially, both core and sheath sections were prepared from the same polymer. Subsequently, the polymer sheath/core combinations were varied to include high density polyethylenes of different molecular weights (M_w) and molecular weight distributions (M_w/M_n). The molecular characteristics of the polyethylenes used are listed in Table I.

(b) Thermal Properties

The melting curves of the extruded samples were determined with a Perkin-Elmer differential scanning calorimeter (CDSC) Model 1B, calibrated by the melt transition of indium. The melt behavior of the samples, ~2.0 mg., was investigated at a heating rate of 10°C. $min.^{-1}$.

(c) Mechanical Properties

The tensile modulus and strength determinations of the extruded samples were preformed at room temperature using an Instron testing instrument, Model TTM. For modulus measurements a strain gage extensometer (10 mm. gage length) was used to measure strains on the deformed samples. The strain rate was 3.3×10^{-4} $sec.^{-1}$. The tensile modulus was determined from the tangent to the stress-strain curve at 0.1% strain. The tensile strength for break was determined at a strain rate 2×10^{-3} $sec.^{-1}$.

RESULTS AND DISCUSSION

The systematic study of the effect of average molecular weight (M_w) and molecular weight distribution (M_w/M_n) on the extrusion behavior has been tested more thoroughly and at lower temperatures than previously. Ultraoriented extrudates (25X) of different M_w and M_w/M_n have been obtained in continuous lengths at temperatures significantly below the melting range. The characteristic features of crystalline state coextrusion may be viewed in Figures 1 and 2 and in Tables II and III. Figure 1 shows the length versus time data for the solid state extrusion of split billets of a single high density polyethylene. Figure 2 shows extrudate length versus time for coextruded composites. Tables II and III list the extrusion rates and the mechanical properties which will be discussed subsequently.

In these studies we used two different families of high density polyethylenes (Alathon and Marlex) since they cover a wide range of different M_w and M_w/M_n.

From Figure 1 and Table II it is clear that the extrusion rate decreases with increasing M_w or decreasing melt flow index. This trend observed separately with each HDPE family, is also observed with the two sets superimposed as shown in Figure 3. Since the extrudates consist of two components, we plotted our results with respect to the average values of the average molecular weight and melt flow index of these components as shown in Table IV. The melt flow index seems to be related to the apparent viscosity on crystalline state

extrusion as discussed below. These were estimated by assuming the law of mixtures and considering the volume fractions of the components. Thus, the extrusion rate varies from 4 cm./min. (at 0.16 GPa) for Alathon 7050 with MFI = 17.5 to 0.06 cm./min. (at 0.23 GPa) for Marlex 6003 with MFI = 0.2. Although variation in M_w/M_n does not influence significantly the extrusion rate, MFI has a more profound effect. This is indicated clearly by examining the molecular characteristics of the studied polyethylenes in Table I and Figure 3. In particular, for polymers D and E with the same M_w the extrusion rate increases with MFI and not with M_w/M_n.

The extrusion rates of the composite extrudates are shown in Table II. It should be noted that the high molecular weight polyethylene was always the sheath component (75% by volume) of the composite billet. It is interesting to note that the viscosity of the semicrystalline state as reflected by the extrusion rate seems to be related to the melt viscosity (MFI) which is a melt state property. The extrusion rate of HDPE single crystal aggregates grown from p-xylene solution is much faster than that of melt crystallized solid plugs[14]. It is believed that the faster rate was caused by the reduced number of tie molecules in the single crystal morphology. According to Keith et al.[15], the number of tie molecules increases with M_w. Thus the observed increase in apparent viscosity on crystalline state extrusion with decreasing MFI is consistent with the above suggestion.

The characteristic feature of the extrusion behavior in the solid state coextrusion of the composite billets is the significant increase in extrusion rate of the high M_w component. In the extreme case of the slow extruding high molecular weight polyethylene F, incorporation of the low M_w polyethylene A as the core resulted in a ten fold increase in extrusion rate. Again, as shown in Figure 3, the extrusion rate of the composite extrudates is a function of the average melt flow index of the component polymers. Although the rates are highly enhanced by the incorporation of a low M_w polyethylene as the core component, they are influenced by the high M_w component which acts as the "bottle-neck" of the coextrusion process under these extrusion conditions. An explanation for the remarkable enhancement in extrusion of the composite billets is offered in the first part of this paper[13]. The object of the present study is mainly the extrusion of ultraoriented fibers with enhanced tensile properties and the effects of the molecular weight and molecular weight distribution. Capaccio and Ward[4] compared the drawing behavior of various commercial polyethylenes and concluded that optimum results are obtained when the average molecular weight is low and the molecular weight distribution is narrow. Barham and Keller[7] on the other hand report that the presence of a low molecular weight component is essential in drawing ultraoriented filaments or films and that it exists in some segregated form. Although the dependence of elastic modulus upon draw ratio is strongly emphasized, it is not clarified which of the two factors i.e. molecular weight or molecular weight distribution,

is the decision and how they affect the tensile properties. However, with the incorporation of a low M_w HDPE as the core segment the extrusion of continuous lengths of the higher M_w HDPE at low extrusion temperature and high EDR is now feasible and these conclusions may be drawn.

Extrusion at the low extrusion draw ratio 12 was also attempted to ascertain the extrusion draw efficiency at low EDR. As it was anticipated, at this low extrusion draw ratio, the tensile modulus of the filaments was independent of the molecular weight but not the tensile strength which increased with molecular weight.

The mechanical property of extrudates of EDR 12 are shown in Table V. From the values of tensile modulus and tensile strength of the single polymers (shown in Table II and Figures 4 and 5), it is clear that there is at least 50% increase in the magnitude of the tensile property as the M_w increases from 59,000 for polyethylene A to 200,000 for polyethylene F. There appears to be no effect of molecular weight distribution on tensile properties as shown by the two polyethylene families. Namely, polyethylene D and E have tensile moduli and strengths of the same magnitudes although these polymers have different molecular weight distributions. Similarly with polyethylene F and G, there is no apparent effect of molecular weight distribution.

The mechanical properties of the composite extrudates may be ascertained by the simple rule of mixtures that applies to a parallel model[16]

$$T = T_c V_c + T_s (1 - V_c) \qquad (1)$$

$$E = E_c V_c + E_s (1 - V_c) \qquad (2)$$

where T = tensile strength, E = modulus, V_c = volume fraction of core component and the subscripts c and s refer to the core and sheath components. As the filament comprising the core of the extrudate is continuous and surrounded by a bonded sheath via melting it is legitimate to use the above equations to describe the mechanical properties. The values of these properties obtained by the above equations are in close proximity with those obtained experimentally.

The results of these studies can be explained in terms of Peterlin's intercrystalline tie molecule model. Accordingly, an increase in molecular weight leads to an increase in the number of tie molecules and the greater number of tie molecules the higher the proportion of extended chains in the non-crystalline phase for a particular extrusion draw ratio. This higher proportion of extended chains enhances the tensile modulus. On the other hand, the tensile strength of the material which results from the crystalline component and the intercrystalline tie molecules, is further increased by the higher number of intercrystalline tie molecules. The latter not only run longitudinally in each microfibril but also laterally between adjacent microfibrils resulting in higher cohesion in the material. These tie molecules prevent microfibrillar slipping.

One salient feature of our solid state coextrusion technique is the feasibility to extrude at high extrusion draw ratio for polyethylene ($\gtrsim$25) and at temperatures substantially below the melting point (~110 - 120^{o}C.). At

such temperatures the deformation process may be more efficient in producing a morphology with a higher proportion of extended chains. This idea is further supported by the results of extrusion at 134°C. and 0.24 GPa[9] and our coextrusion results of low draw ratio (12X) at 110°C. and 0.10 GPa. In either case, the conditions do not favor the attainment of efficient extension of molecular chains between intercrystalline tie molecules. In the first case there is excessive thermal energy causing relaxation of the amorphous phase and therefore amorphous disorientation. In the second, the low extrusion draw does not cause sufficient deformation to produce a morphology with a high proportion of extended chains. Consequently, the effect of molecular weight on modulus cannot be detected satisfactorily under these conditions. Finally, the structure of the coextrudates was also studied by thermal analysis. Figure 6 shows the DSC melting curves of single polymer extrudates and coextrudates obtained at a heating rate of 10°C./min. Single polymer extrudates exhibited sharp single melting peaks which were significantly higher (~7°C.) than the original billets (Figure 7). In contrast, all the coextrudates exhibited double melting peaks irrespective of heating rate (2.5-20°C./min.), indicating that they were not caused by a reorganization during heating but were coexistence of two different morphologies.

Melting curves of HDPE extrudates exhibiting two melting peaks have been reported on irradiated[17], nitric acid etched[18,19] and annealed[20] samples. In all cases, the two peaks were attributed to different morphological components. In order to clarify the two melting peaks, the coextrudates were

separated into sheath and core components and their melting curves were recorded at heating rate 10°C./min. One example is shown in Figure 7. It is clearly seen that the melting peak temperature of the sheath (high M_w HDPE component) is higher than that of core (low M_w HDPE), and that the double melting peaks in coextrudates were caused by the two components.

Another feature to be noted is that the sheath and core peaks are sharp and equivalent to those of each single polymer extrudate shown in Figure 6. Furthermore, the area of lower melting peaks is about one fourth of the total peak area, the ratio corresponding to the volume fraction of the two polyethylene components in the initial billet. These facts indicate that the sheath and core polyethylene components were extruded at the same rate and maintained the original geometrical arrangement of definite sheath and core even after coextrusion. Although the melting peak temperature alone cannot be used as a sound criterion for the efficiency of draw, these DSC results suggest high efficiency of draw for both of sheath and core polyethylene components.

CONCLUSIONS

The mechanical properties of cocylindrical composite fibers extruded by crystalline state coextrusion have been studied for various commercial high density polyethylenes of different molecular weights and distributions. The method is unique in producing ultradrawn fibers (EDR 25). The results of this study show that the extrusion rate does not depend on the molecular weight distribution and is directly related to the average molecular weight or melt flow index of the component polyethylenes. Similarly, the mechanical properties increase with the average molecular weight but do not depend on the molecular weight distribution. The mechanical property results can be described by the intercrystalline tie molecule model, i.e. the fraction of continuous crystals, and ascertained by the law of mixtures for a parallel model. The efficiency of drawing by coextrusion was evaluated by thermal analysis.

ACKNOWLEDGEMENT

The authors express their appreciation to the Office of Naval Research for financial support.

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TABLE I

Molecular Characteristics of (studied)
High Density Polyethylenes

Polymer Designation	M_w (x 10^{-3})	M_w/M_n	Melt Flow Index
A	59	2.96	17.5
B	92	3.54	2.8
C	147	4.43	1.0
D	$\leq$110	<7	6.5
E	$\leq$110	13-20	5.0
F	=200	7-13	0.3
G	$\leq$250	13-20	0.2

TABLE II

Extrusion Rate and Tensile Properties for
Individual High Density Polyethylene Extrudates

High Density Polyethylene Grade	Extrusion Rate (cm./min.)	Tensile Strength (GPa)	Tensile Modulus (GPa)
A	4.0*	0.37	28
B	1.0	0.45	25
C	0.15	0.52	31
D	1.7	0.49	23
E	0.8	0.48	23
F	0.06	0.55	45
G	0.05	0.47	45

*Extrusion performed at 0.16 GPa pressure.

TABLE III

Extrusion Rate and Tensile Properties of Composite
High Density Polyethylene Extrudates

Designation (sheath/core)	Extrusion Rate (cm..min.)	Tensile Strength (GPa)	Tensile Modulus (GPa)
B - A	2.5	0.43	31
C - A	0.2	0.48	26
D - A	2.7	0.44	32
E - A	2.5	0.34	26
F - A	0.5	0.6	36
G - A	0.25	0.51	38

TABLE IV

Average M_w and MFI of Coextruded
High Density Polyethylenes

Polymer Designation*	M_w ($\times 10^{-3}$)	MFI
B - A	83.7	6.5
C - A	125.0	5.1
D - A	97.2	9.2
E - A	97.2	8.1
F - A	202.2	4.6
G - A	202.2	4.5

*Sheath/core assignment.

TABLE V

Tensile Properties of Individual and Composite
High Density Extrudates at EDR 12

Polymer Designation	Young's Modulus (GPa)	Tensile Strength (GPa)
A	9	0.25
B	11	0.39
C	12	0.40
F	10	0.45
B - A*	9	0.47
C - A*	9	0.38
F - A*	10	0.40

*Sheath/core assignment.

FIGURE CAPTIONS

Figure 1: Extruded Filament Length versus Time Curves for Individual Polyethylenes. Extrusion Conditions = T_{ex} = 120°C., P_{ext} = 0.23 GPa, EDR = 25, No Lubricant.

Figure 2: Extrudate Length versus Time for Polyethylene Composite Extrudates. Extrusion Conditions: T_{ext} = 120°C., P_{ext} = 0.23 GPa, EDR = 25, No Lubricant. The sheath-core order indicates the geometrical arrangement of the polyethylene phases.

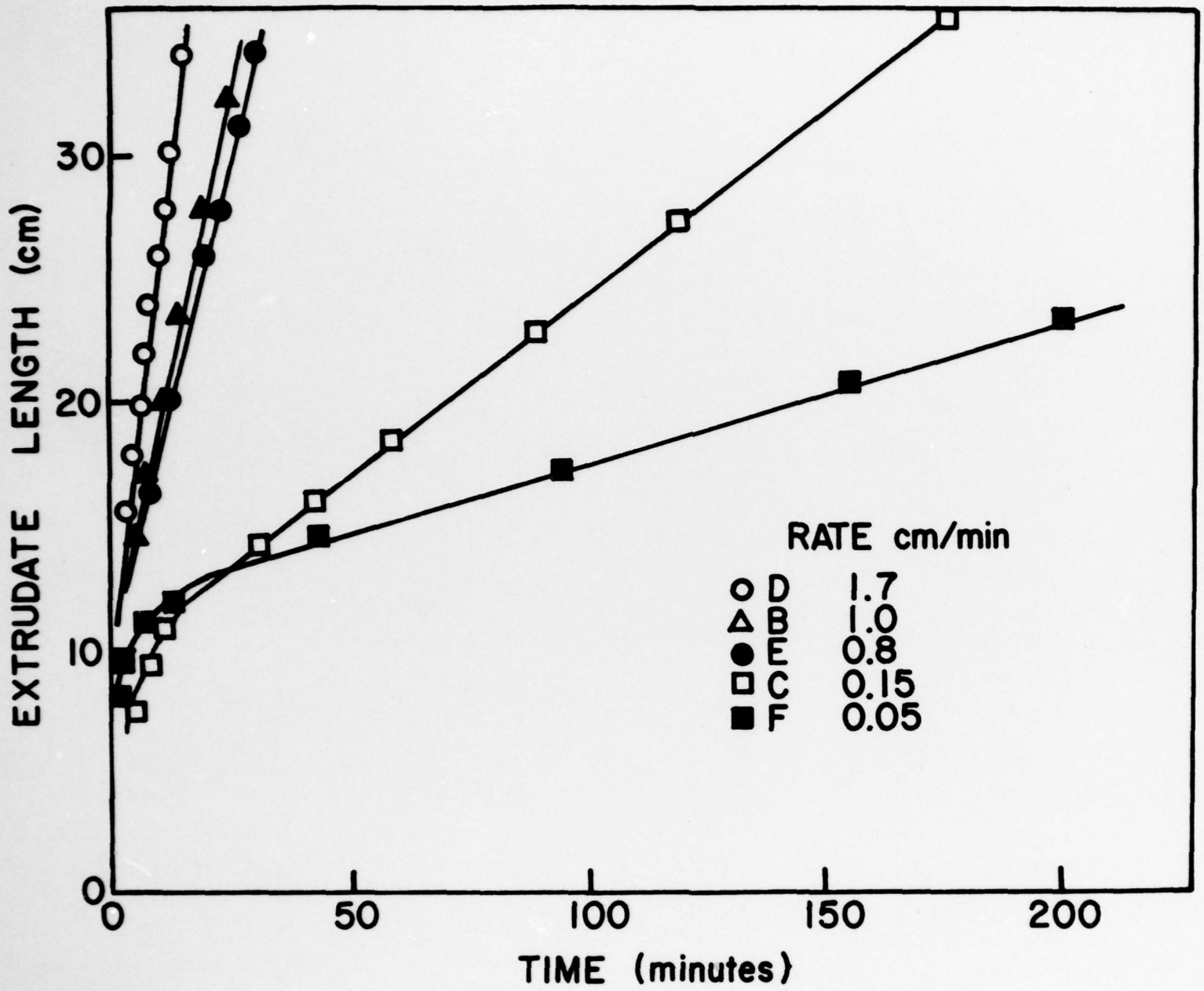
Figure 3: Extrusion Rate versus Melt Flow Index for Single and Composite Polyethylene Extrudates (EDR = 25). For composite extrudates the MFI values represent the average value for the two components on the basis of their volume fractions.

Figure 4: Tensile Modulus as a Function of Molecular Weight for Individual and Composite Polyethylene Extrudates. For composite extrudates, the M_w represent the average value for the two components on the basis of their volume fractions.

Figure 5: Tensile Strength as a Function of Molecular Weight for Individual and Composite Polyethylenes. For composite extrudates, the M_w represents the average value for the two components on the basis of their volume fractions.

Figure 6: Melting Thermograms of Single Polyethylene Components (a) and Coextrudates (b); Heating Rate 10°C./min.

Figure 7: Melting Thermograms of C - A Coextruded Fiber and its Sheath and Core Components. Thermograms of the initial preformed billet components are also included.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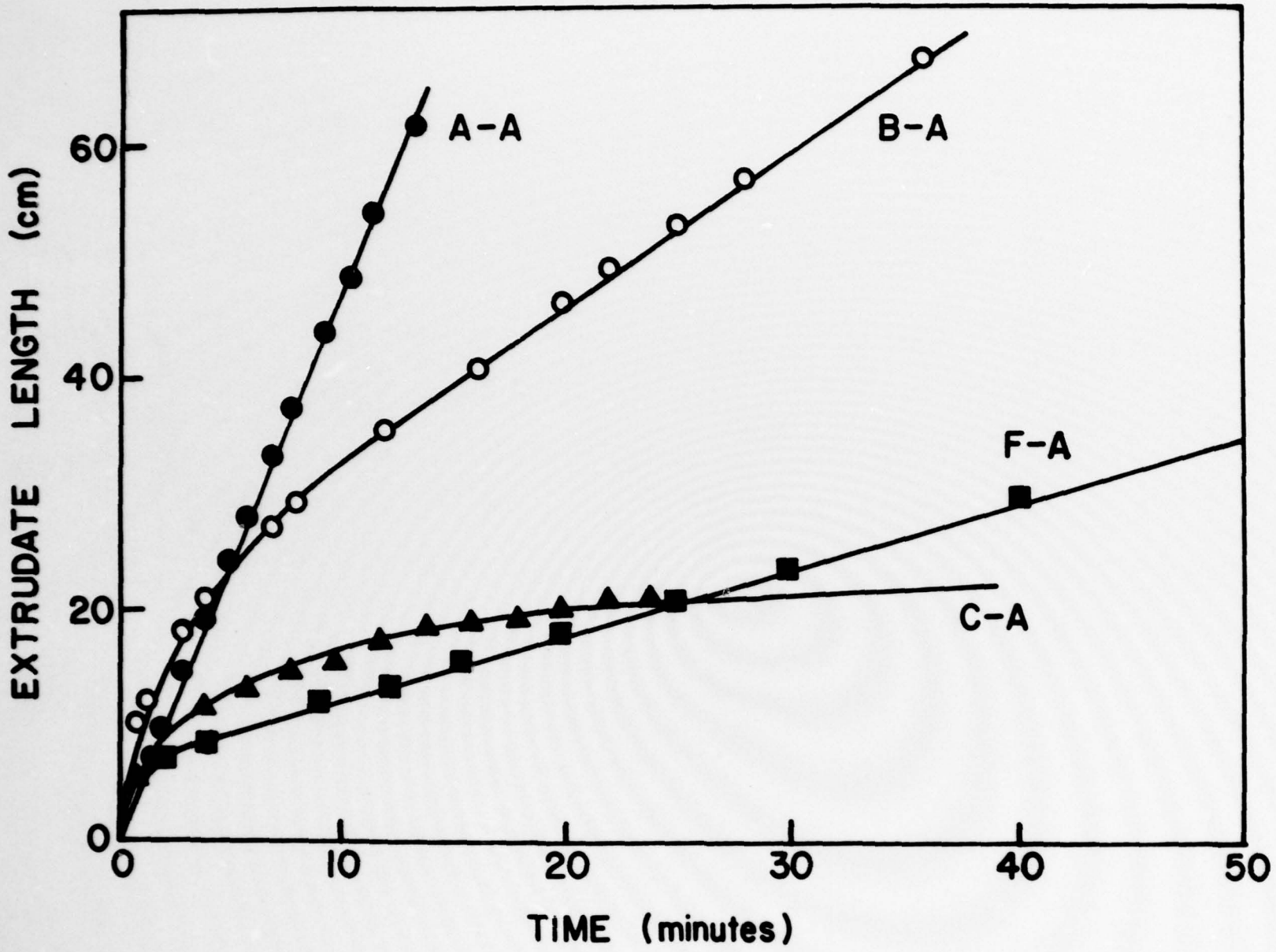
C 0.15
F 0.05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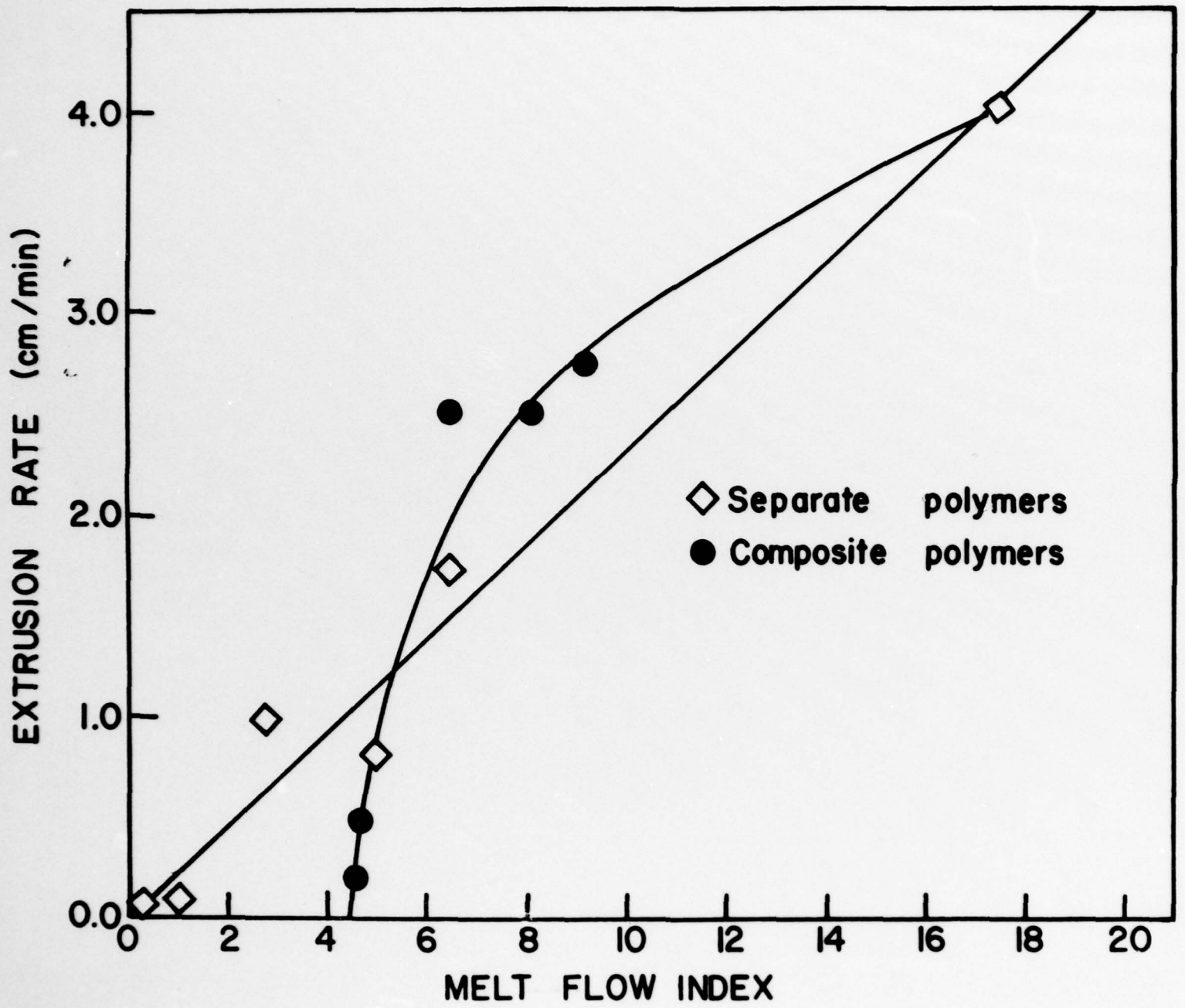
F-A
C-A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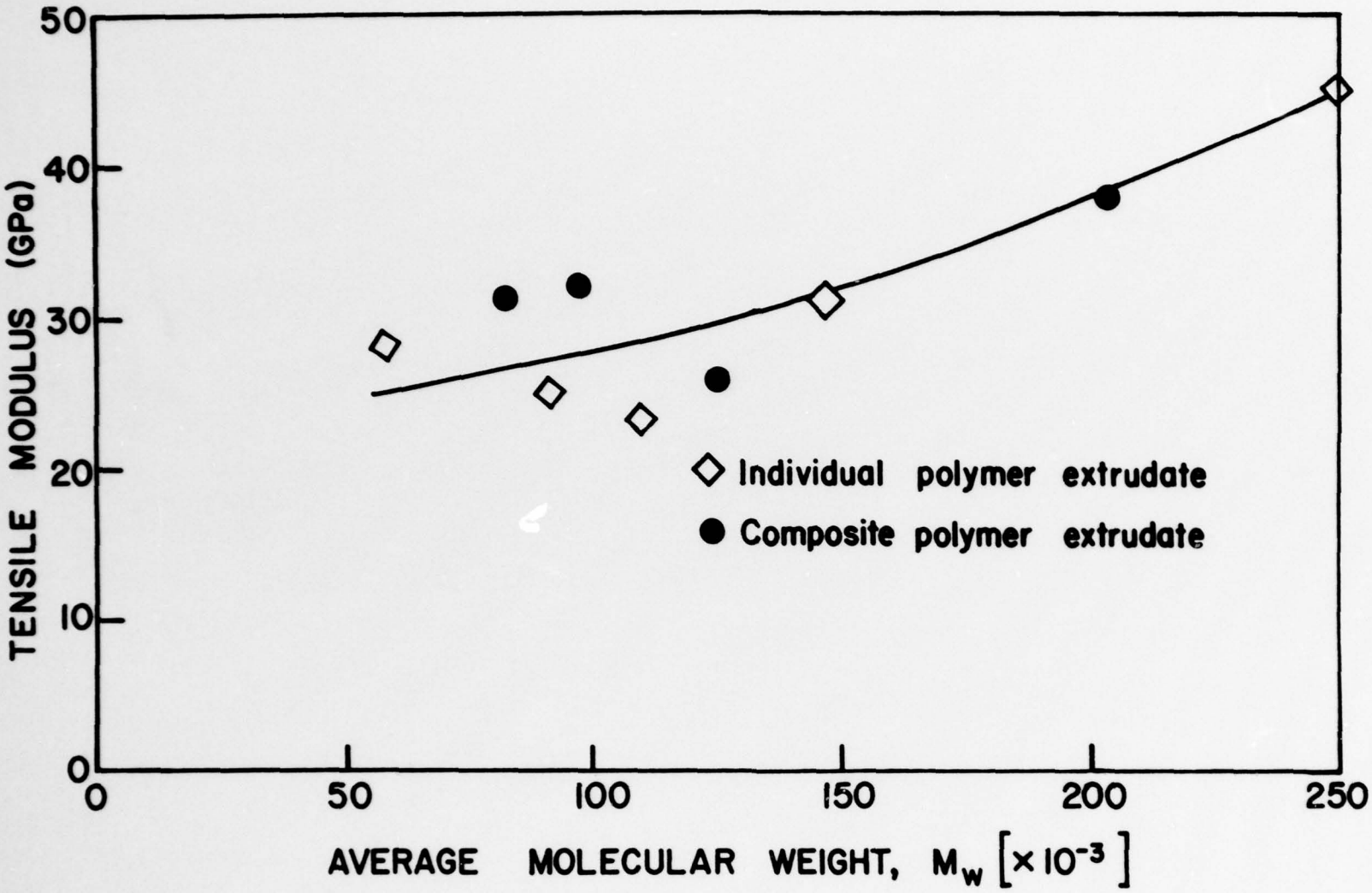
18
20
MELT FLOW INDEX
Separate polymers
Composite polymers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200
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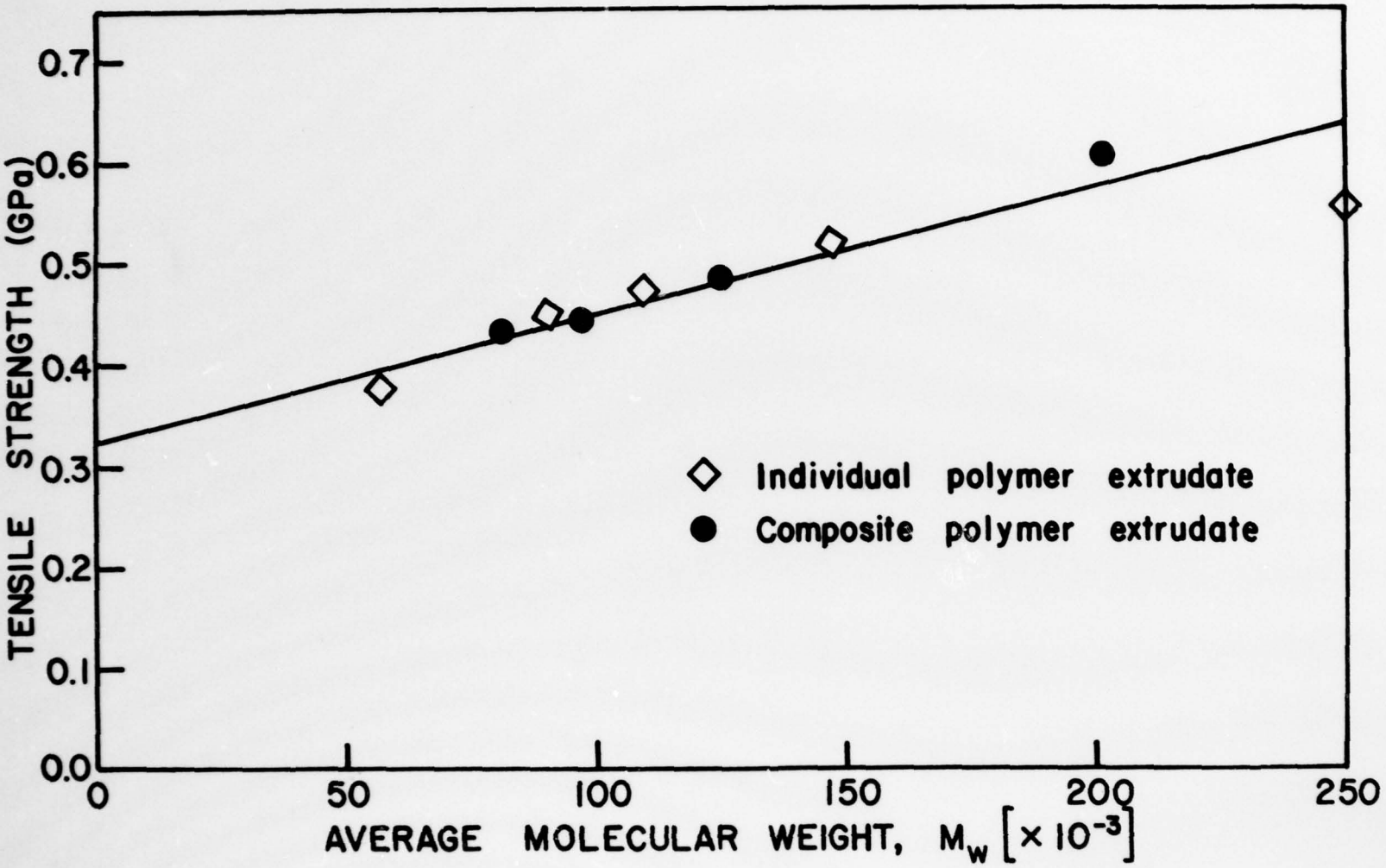
Individual polymer extrudate
Composite polymer extrudate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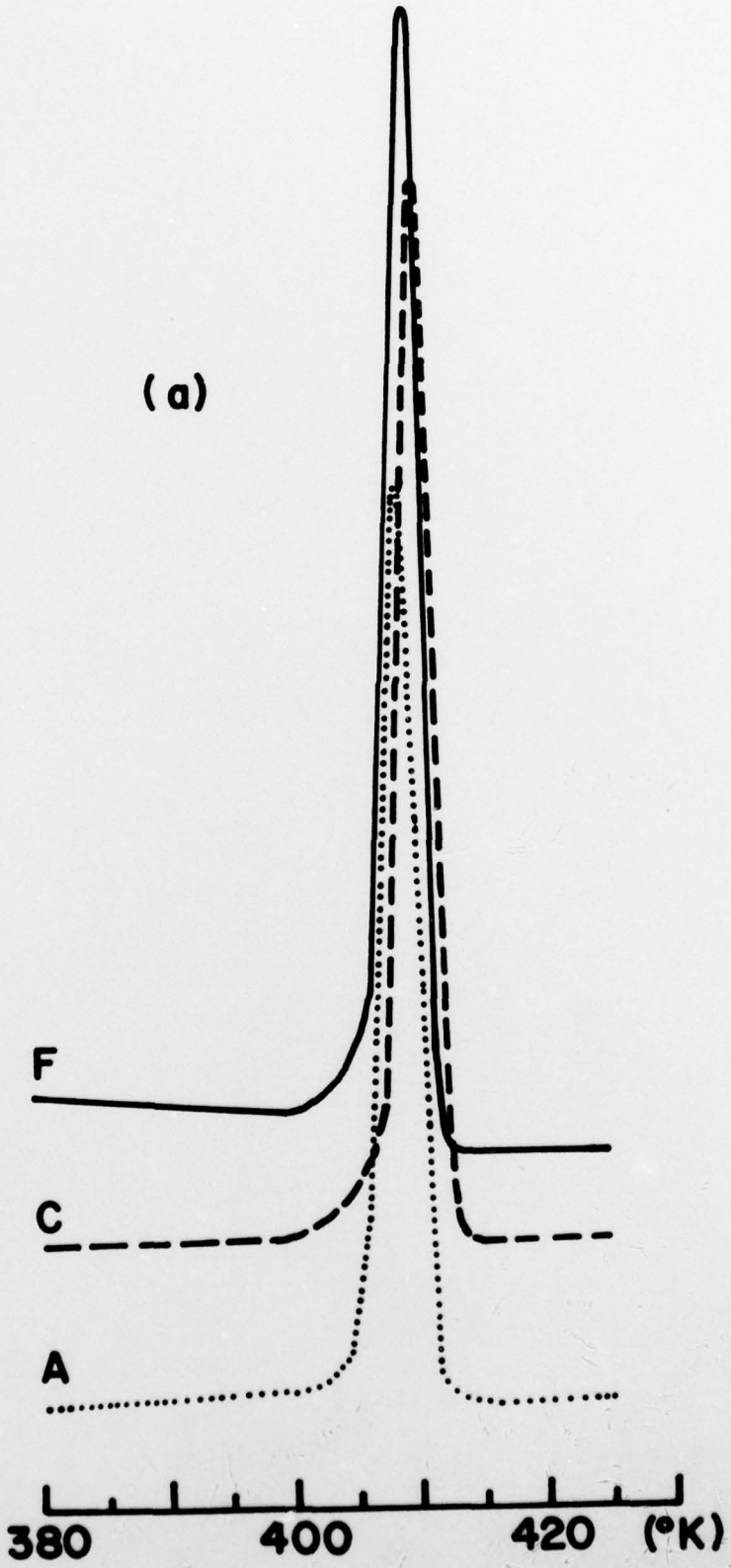
AVERAGE MOLECULAR WEIGHT, M_w [$\times 10^{-3}$]
Individual polymer extrudate
Composite polymer extrudate


(a)
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C
A
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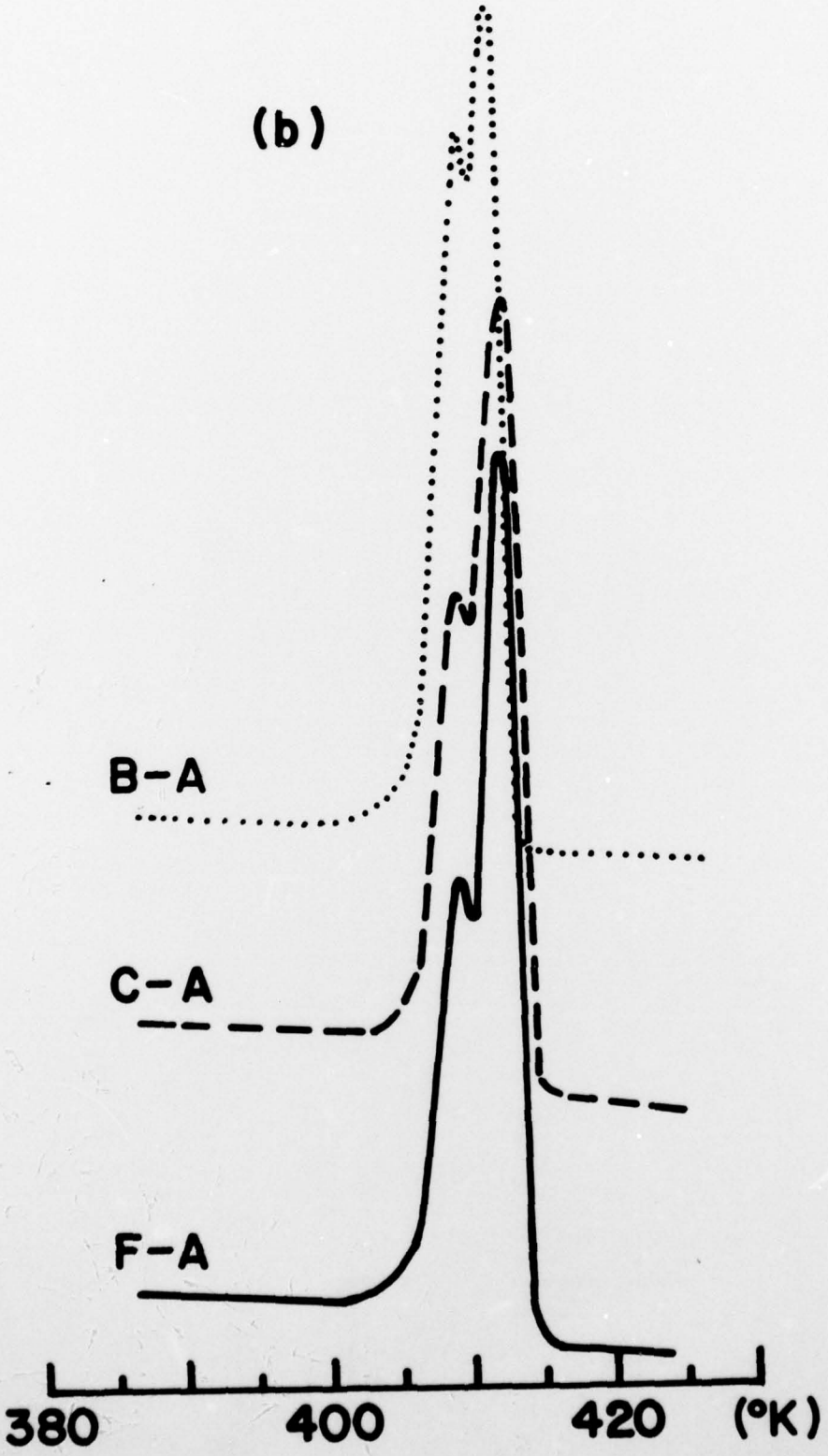
400
420
(°K)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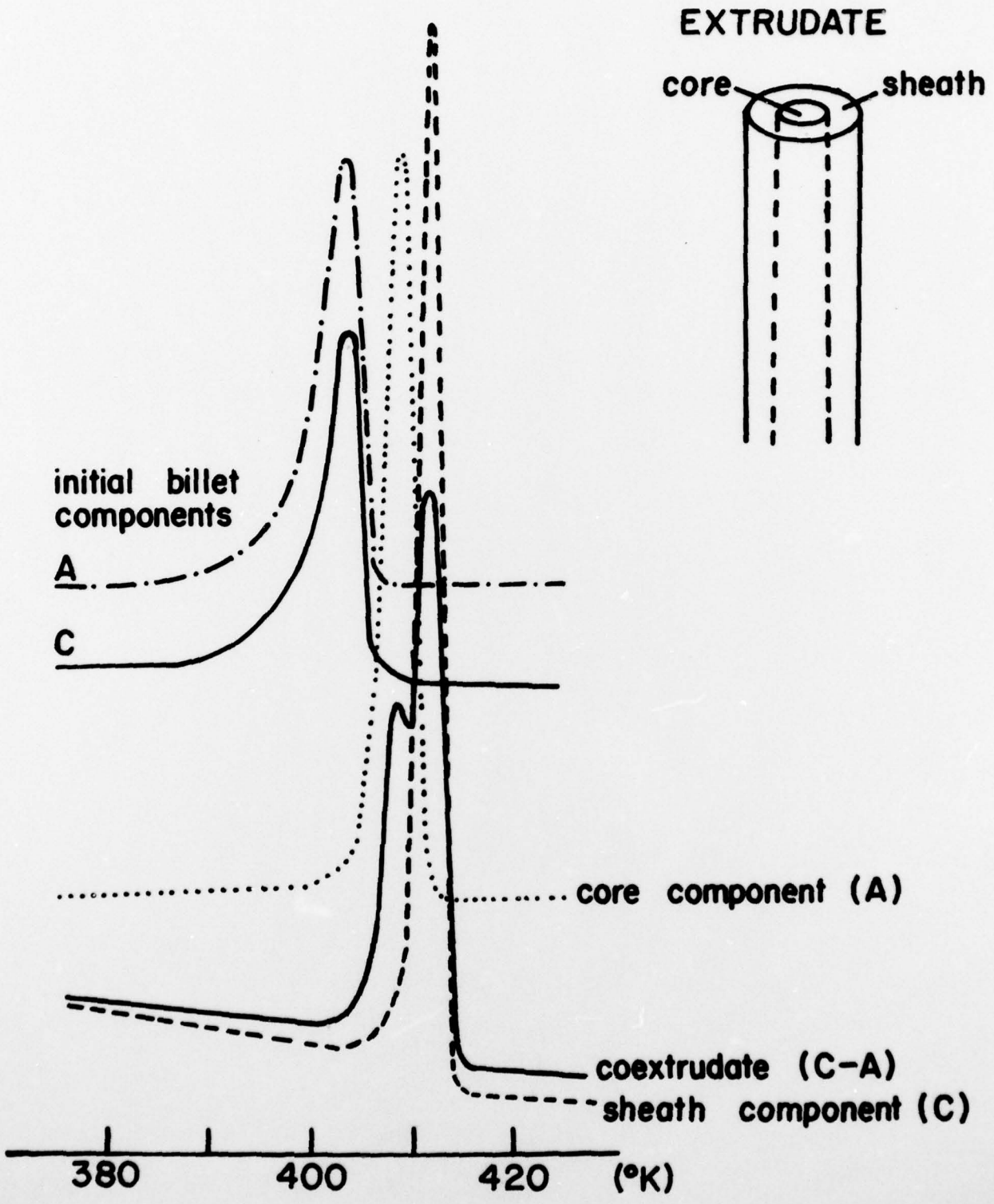
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initial billet components
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coextrudate (C-A)
sheath component (C)
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Unclassified

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REPORT DOCUMENTATION PAGE	READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER: Technical Report No. 11	2. GOVT ACCESSION NO. / 3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle): Solid State Coextrusion of High Density Polyethylene. II. Effect of Molecular Weight and Molecular Weight Distribution	5. TYPE OF REPORT & PERIOD COVERED: Interim
	6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s): Anagnostis E. Zachariades, Tetsuo Kanamoto and Roger S. Porter	8. CONTRACT OR GRANT NUMBER(s): N00014-75-C-0686
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Polymer Science and Engineering, University of Massachusetts, Amherst, Massachusetts 01003	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: NR 356-584
11. CONTROLLING OFFICE NAME AND ADDRESS: ONR Branch Office, 666 Summer Street, Boston, Massachusetts 02210	12. REPORT DATE: October 16, 1978
	13. NUMBER OF PAGES: 29 (incl. tables and figures)
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)	15. SECURITY CLASS. (of this report): Unclassified
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

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solid-state extrusion, ultradraw, high-density polyethylene, molecular weight distribution, self-reinforced, composites, volume fraction, mechanical properties

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The recently developed technique of solid state coextrusion for ultradrawing semicrystalline thermoplastics has been applied in the preparation of self-reinforced high density polyethylene extrudates. The extrudates consist of definite core and sheath phases composed of different molecular weights (Mw) in the range of 60,000 - 250,000 and different molecular weight distributions (Mw/Mn = 0.3 - 20). Cocylindrical billets of two different phases were prepared for extrusion by inserting a polyethylene rod within a tubular billet

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of a different high density polyethylene followed by melting the two phases to obtain bonding between them. The billet was then split longitudinally to increase extrusion speed followed by extrusion at 120°C, 0.23 GPa and extrusion draw ratio 25. Thus it was possible to produce extrudates of high tensile modulus (45 GPa) and strength (0.66 GPa) at a rate near 0.7 cm./min. In general, the tensile properties of the extrudates increased with average molecular weight and were insensitive to the molecular weight distribution of the two phases.

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